APPENDIX C
PROJECT-SPECIFIC OPERATING PROCEDURE
FOR OPSIS UV-DOAS CALIBRATION AND QC CHECKS FOR BENZENE

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1.0 Scope and Applicability

The purpose of this project-specific operating procedure (PSOP) is to instruct field personnel in performing benzene calibration and calibration checks for the OPSIS UVDOAS Spectrometer. This PSOP is applicable only to the calibration of the OPSIS UVDOAS Opto-Analyzer AR 500 for benzene. This PSOP also includes procedures for OPSIS set up and system checks required for the calibration procedures.

2.0 Summary of Method

This PSOP describes the method used to calibrate and verify the calibration of the OPSIS UVDOAS instruments for measurement of airborne benzene using a specific measurement configuration. The method involves setting up the optical calibration bench, optimizing the source light throughput, inserting gas calibration cells, and calibrating the analyzer. There are two primary methods described:

8-point Span/Offset Calibration: This method is used to calibrate the OPSIS system for benzene measurements

4-point Calibration Check: This method is used to verify the calibration of the OPSIS system for benzene measurements

Chapter 5 of the OPSIS Opto-Analyser AR-500 Hardware Operating manual and OECA/OCE's "Standard Operating Procedure for UV OPSIS calibration" December 2000 can be consulted to provide supplemental information in this PSOP. General information of use of OPSIS analyzer software can be found in "OPSIS Analyzer Software User's Guide, Version 7.2". The OPSIS "Qaulity Assurance and Quality Control using Opsis Analyser for Air Quality Monitoring" Version 1.4, should also be consulted as general reference for calibration, operational check and quality assurance procedures for the OPSIS analyzers.

Notes:

(1) The analyzer will be calibrated in units of ppbV, and corrected for Standard Temperature and Pressure for consistency with the NIST-traceable reference gas standard certification on a volumetric basis. Field measurements will be conducted in units of actual ug/m^3 for consistency with the DIAL measurements of mass flux.

(2) The USEPA takes certain quality assurance steps not specified in the OPSIS manuals due to the use of the instrument for field measurements. Note that on the hard copy Calibration Forms (attached as appendices H1 through H4), there are entries for the lamp ID and fiber ID. The lamps used in the telescope and calibration bench may change at different rates over long periods of time due to difference in usage. Differential lamp usage can result in small changes in the benzene offset between the calibration bench and the emitter telescope. Therefore, for benzene measurements, a field check for the benzene offset is conducted by USEPA prior to deployment to ensure the lamp reference spectra emitted by

the telescope is comparable to the calibration bench. A small offset adjustment, normally in the range of 1 or 2 ppbV, can be made to account for differential lamp aging. A record of the lamp ID used during calibration and field measurement is therefore necessary. Additionally, USEPA uses the same fiber from the calibration receiver to the analyzer as is used during field measurement to minimize the variables during calibration. Finally, the frequency of calibration is increased due to the transportation of the analyzer or necessities of the project (see Table 2).

3.0 Definitions

Atmospheric Pollution Prevention and Control Division (APPCD)
Infrared (IR)
Light Emitting Diode (LED)
Mercury (Hg)
Normal Temperature and Pressure (NTP)
Office of Enforcement and Compliance Assurance/Office of Compliance (OECA/OCE)
Project-Specific Operating Procedure (PSOP)
Quality Assurance Project Plan (QAPP)
Standard Operating Procedure (SOP)
Ultraviolet (UV)
Ultraviolet Differential Optical Absorption Spectrometer (UVDOAS)
United States Environmental Protection Agency (USEPA) or (EPA)

4.0 Health and Safety

Personnel must be familiar with the requirements set forth in the Health and Safety SOP developed specifically to address health and safety issues associated with operation and maintenance of the OPSIS UVDOAS monitoring system and all ancillary equipment and/or activities. This PSOP also requires the use of hazardous chemicals including mercury. See EPA OECA/OCE's "Standard Operating Procedure for Health and Safety", December 2000 and APPCD's "OPSIS Safety Protocol", November 2005.

5.0 Cautions

Appropriate protective eye wear must be worn by operators of the OPSIS UVDOAS monitoring system anytime the equipment is on and the operator is in the proximity of the beam path.

Calibration of the UVDOAS involves use of a calibration bench which minimizes direct exposure to IR and UV light; however, personnel should avoid looking directly into the light source beam and should wear protective eye wear anytime the light source is in use.

6.0 Personnel Qualifications

Personnel conducting the instrument calibration procedures should have a working knowledge of the OPSIS UVDOAS monitoring system and be able to comfortably execute the physical activities associated with setting up the instrumentation (i.e., lifting and moving the calibration bench, lifting the analyzer and computer module from storage locations to the calibration area, etc.). Personnel should be familiar with the EPA OECA/OCE's "UVDOAS Field Operation SOP" December 2005 can be used in conjunction with this Calibration PSOP.

7.0 Apparatus and Materials

7.1 OPSIS gas calibration cells in the following lengths in mm: 100.6, 248.8, and 501.5 are to be used with a NIST-traceable benzene calibration gas standard of approximately 50 to 200 ppmV of benzene, balance air or nitrogen, certified accuracy of ± 2%.

7.2 OPSIS UVDOAS Opto-Analyzer: The AR500 used to perform field measurements for this PSOP is identified as OCE System # A00015.

7.3 OPSIS Calibration Bench: The OCE system bench is used for all calibration work.

7.4 Xenon Calibration Lamp: A Type B lamp identified as "R8" is used for all calibration work.

7.5 Lamp Power Supply: The OCE xenon lamp power supply is used for all calibration work.

7.6 Lux Light Meter: The OCE lux meter (Hagner EC1) is used for all work.

7.7 Fiber Optic Cables: Fiber optic cables used during field measurements are used to connect system components. Fibers are labeled for use as follows:

- 2m fiber OF60S 942 connects Xenon lamp to cal bench
- 2m fiber OF60S 944 connects Hg reference lamp to analyzer
- 5m fiber CALR7-02 is dedicated to OCE analyzer

7.8 OPSIS Hg Reference Lamp: The OCE Hg reference lamp is used for all work.

7.9 Climatronics Tacmet II, Ser. No. 187, for analyzer temperature and pressure logging during calibration.

8.0 OPSIS Set Up and Operational Checks

This section describes the basic operational procedures and checks that are performed on the OPSIS analyzer to ensure that it is functioning properly after the unit has been moved or started prior to calibrations and/or measurements. The schedule of various types of calibration and operational checks for this PSOP is shown in Table 2.

8.1 Cabling Check:
Check all electrical power connections (i.e., computer, monitor, dry air pump, backup power supply) before plugging the analyzer into the external power source.

8.2 Calibration Bench Set Up:
Set up the calibration bench and plug in the calibration lamp as shown in Figure 2 and Figure 5. Start the calibration lamp and allow it to stabilize for at least 30 minutes. (Note: The center of the optical bench may be supported with two wooden wedges or equivalent to prevent optical path sag distortion when calibration cells are placed in the bench. Also, no UV filter is used for calibration).

8.3 Analyzer Connections:
Connect the analyzer to the external power and start the dry air purge for the system using the manual off/on switch on the lower front of the console. Allow the analyzer to purge with dry air for 10 minutes.

8.4 Fiber Connections:
Connect the receiver telescope fiber optic cable from the calibration bench receiver to the analyzer using the specific 5 m labeled fiber. A specific labeled 2m fiber is used to connect the calibration lamp to the bench emitter (see Section 8.7 and Figure 1). Turn on the analyzer. (Note: Always connect the fiber optic cable and check major electrical connections <u>before</u> starting measurements. Handle fiber optic cables with great care. Do not twist or bend cables sharply. Do not touch the exposed fiber optic filament to any surfaces.)

8.5 System Check:
Perform system checks after the system has been activated. If measurements were collected immediately prior to the system check, abort the measurements software by pressing the escape key (i.e., [Esc]) on the analyzer keyboard, followed by pressing the function key [F5] to begin the check. The analyzer will automatically execute several system checks for:

- Rotating disk stability
- Grating positioning
- Detector system performance

Perform the system check and record the first set of values (P1-P4) in the analyzer log book and/or calibration Forms (These values can be used on the Forms H1,H2, and or H4). Normally, the Wheel Speed and all P parameters will be reported as "OK." However, a value may be reported as "Fail." In this case, conduct the system check again. The error should self-correct and this result should be entered on the form. Parameter P4 can be adjusted. Although OPSIS allows a range of ±20, USEPA prefers to narrow the range to ±5. Answer Yes [Y] when prompted to adjust the P4 factor and conduct the check again. Record the final results in the logbook and/or calibration forms. DO NOT adjust P4 if P3 has failed. In such cases, conduct the system check again, as noted above.

After completing the system checks, bring the system back into the analyzer root menu by pressing the escape key (i.e, [Esc]).

9.0 <u>Wavelength Precision Check</u>

The wavelength precision test verifies that the wavelength range seen by the analyzer is identical to the range specified during manufacture. The Hg spectrum from the OPSIS Hg reference lamp is used as the wavelength standard for most of the materials monitored by the OPSIS UVDOAS system (including atmospheric benzene). The precision of the grating position and, therefore, the wavelength accuracy is evaluated by comparing the spectrum from a current Hg reference lamp analysis to the one recorded when the instrument originally met manufacturing specifications. To execute the Wavelength Precision Check, perform the following procedures:

9.1 Software Navigation:
Exit analyzer measurement mode, if necessary, by pressing the escape key (i.e., [Esc]), pressing the function key [F3] for calibration procedures, pressing the function key [F6] for advanced calibration procedures, and pressing the function key [F1] for the check wavelength precision function.

9.2 Hg Light Source Connections:
Remove the calibration bench fiber optic cable from the analyzer, and connect the the 2m Hg lamp fiber optic cable between the Hg reference lamp and the analyzer. Turn the lamp on by pressing the black Lamp Start button. The green LED on the lamp housing should light up indicating the lamp has been energized and is operating.

9.3 Hg Lamp Warm-up:
Allow the Hg lamp to warm up for at least 5 minutes.

9.4 Hg Gas Selection:

For this PSOP, select "Benzene" as the only gas for calibration from the analyzer calibration menu using the arrow key and space bar, then press the enter key.

9.5 Wavelength Precision Test:
Specify 30 seconds for the time the spectrum should be recorded. Press the enter key again to start measuring the Hg spectra. Record the results in the log book and or calibration forms.

Results are shown by the analyzer data system in two ways: graphically or numerically. The graphical presentation shows an overlay of the original spectrum and the current spectrum. Press the escape key [i.e., Esc] and calculate the numerical channel shift and the accuracy factor. An accuracy factor of 50% or higher is acceptable. The channel shift correction calculation must not exceed ±5 channels. If the channel shift correction is greater than 5 channels, a correction can be made following the on-screen instructions. Always repeat the wavelength calibration check after making an adjustment to ensure the instrument is operating within proper specifications.

10.0 8-point Span/Offset Calibration for Benzene Measurements

This procedure describes the 8-point calibration (with non-linear correction) of the OPSIS system for atmospheric benzene measurements. Procedures described here supplement the reference calibration performance procedures in Section 5 of the OPSIS Opto-Analyzer Hardware Operating Manual and Quality Assurance Manual (particularly section 5.7.5, Non-Linear correction). All calibrations and calibration checks are performed with analyzer-specific fiber optics specified in Section 8.

The OPSIS analyzer response is calibrated using calibration cells containing a reference gas standard from a reputable vendor. The expected response of the analyzer (measured benzene concentration) is a function of the calibration gas cell length, the calibration gas concentration, and the chosen path length. For this project the reference path length for calibration shall be 250 m. The minimum linear span range shall be 0 to 500 ug/m^3 of benzene for a 250 m path.

Good record keeping is essential and must be followed by the instrument operator. An analyzer-specific log book and hard copy calibration sheets must be maintained together with the instrument. For the procedure, the OPSIS Multipoint Span/Offset Calibration Form (Appendix C1) and the Multipoint Span/Offset Calibration Worksheet (Appendix C2) are used to record all calibration data.

10.1 Instrument System Checks and Calibration Bench Setup:

Follow the procedures of Sections 8 and 9 performed prior to the 8-point Span/Offset Calibration.

10.2 Calibration Form Preparation:
Retrieve a blank copy of the OPSIS Multipoint Span/Offset Calibration Form. (Reference Appendix C1) and the Multipoint Span/Offset Calibration Worksheet (Appendix C2).

<u>Multipoint Span/Offset Calibration form</u>:

· Fill-in the title section (top block) with analyzer and site information

· Fill-in the Calibration Setup Data
  Strike default cell lengths and replace with:
  L_{01} = 0.1006m
  L_{02} = 0.2488 m
  L_{03} = 0.5015 m
  Fill-in the remainder of blocks as follows:
  <u>Temperature</u>: This will be logged by the analyzer
  <u>Pressure:</u> this will be logged by the analyzer
  <u>Light Int.</u> (from analyzer in %): <u>Lux</u> 1500 to 1,900 (See 10.5)
  <u>GG 400?</u>: no,
  <u>LF215/220?</u>: no

· Fill-in the Initial Analyzer Data Calibration Setup Data
  <u>Monitoring Path Length, L</u>: 250 m (see Table 1)
  <u>Reference ID</u>: Current Reference date (eg 051107)
  <u>Emitter Lamp ID</u>: B7
  <u>Span Setting (existing)</u>: Record the current span setting
  <u>Offset Setting (existing)</u>: Record the current offset setting

· Fill-in the Preparations block:
  <u>Cal. Measurement time</u>: 56 seconds
  <u>Initial System Name:</u> N/A
  <u>System Name Changed:</u> N/A
  <u>Cal. System Name "SPAN.CAL"</u>: N/A

· <u>Normal Path Measurement Time:</u> 56 seconds

· Fill-in the System Check block: Use data from procedure 8.5

· Fill-in the Calibration Lamp CA 004 test: Use data from section 9.5

<u>Multipoint Span/Offset Worksheet</u>

- Fill-in the title section including the active reference file

- Fill-in the title Fiber and Lamp ID information

The Multipoint Span/Offset Calibration Worksheet will be used to record the raw data to be averaged for the final calibration record (Multipoint Span/Offset Calibration form).

10.3 Calibration Lamp Output:
Measure the calibration lamp output by decoupling the 2 m fiber from the calibration emitter and connecting it to the Lux meter. Record the calibration lamp output on in log book for future reference. Expect Lux values in the range of 9,000 to 30,000 depending on lamp age. Reconnect the 2m fiber the calibration emitter (reference Figure 1).

10.4 Calibration Bench Optimization:
Optimize the calibration bench alignment: Disconnect the 5 m fiber from the analyzer (if not already) and connect to the lux meter to measure the amount of light coupled through the calibration bench and 10 m fiber (see Figure 3). Optimize the alignment of the calibration bench through iterative adjustment of the mirror positioning and fiber positioning systems located in the calibration bench emitter and receiver components (see Figure 1). Obtain the a maximum light level of approximately 5,000 lux. For this procedure, ensure that that the bench tray is free of objects.

10.5 Set Calibration Light Level:
After the calibration bench has been aligned, reduce the light level measured at the 10 m fiber to 1500 to 1,900 lux by defocusing the receiver fiber stage. This will establish a standard baseline light intensity level for calibration procedures. This light intensity level, once the calibration cells are installed, is similar to that which will be encountered in the field.

10.6 Verify Light Spot Quality:
Remove the 10 m fiber from the Lux meter connection and inspect the quality of the light spot by directing the fiber output to a white piece of paper or business card held approximately 10 to 15 cm away. The light spot emanating from the fiber should be smooth without many noticable dark spots or rings in it. If the light spot does not appear uniform, place a loose- radius bend in the fiber to mix the guided optical modes, this should produce a uniform spot. Secure the fiber in this configuration using tie wraps. Take care not to bend the fiber at a sharp radius (not less than about 3 inches) as this could cause damage to the fiber.

10.7 Fiber Connection:
Connect the 5 m fiber to the analyzer.

10.8 Xenon Lamp Reference Check:
Refer to Section 12.0.

10.9 Prepare Instrument and Calculate Expected Response:
The following procedure will generate and record the 8-point Span/Offset Hg Calibration data:

10.9.1 Measurement Parameter Setup:
Set up the measurement menu using the calibration parameters identified in Table 1. See the Analyzer Field Operation SOP for detailed instructions on analyzer data system operation. Go to the **Change span/offset** menu under **Calibration**, make a note of the existing span and offset values for path one, and set span to 1.0 and offset to 0.0 for the 8-point Span/Offset Calibration.

10.9.2 Temperature and Pressure Correction Setup:
Go to the **Path specifications** menu under **Measurement setup**. Ensure the the NTP (temperature/pressure correction) is set to "25C." The standardized concentration of benzene is used for calibration for consistency with the reference gas standard.

10.9.3 Note Calibration Start and Stop Time:
Since the analyzer will be operating in normal measurement mode during calibration, it is critical not to confuse normal measurement values with calibration values. Make sure to note the start and stop times for all calibration work in the analyzer log book so that calibration data can be properly segregated from actual measurement data during data validation and archiving .

10.9.4 Check and Record Lux Level:
Ensure that the reduced lux level of (default 1500 - 1900 lux) set in 10.5 is accurate and properly recorded in calibration setup data of the calibration form.

10.9.5 Insert Calibration Cells:
Place the three cells in the bench and fill the cells with the reference gas standard.

10.9.6 Record the Expected Analyzer Response (X):
Calculate the expected response value of the instrument (X) for each of the cell combinations in 10.9.5 and record the value in column 4 of the Multipoint Span/Offset Calibration Form adjacent to its respective total Cal. Cell Length (L_c). It is noted that (X) is based on (and equivalent to) a length corrected analyzer response (C_c') which is modified by the stored measurement path length (L). The measurement path length (L) is automatically present in all concentration calculation so must be decoupled appropriately. An example calculation follows:

$X = C_c' = C_c \times (L_c/L)$;

Where: C_c' = Length Corrected Analyzer Response (in ppbV)
C_c = Hg Cell Concentration (in ppbV) ....
L_c = Total Cal. Cell Length
L = Measurement path length

Repeat this calculation for all entries and record appropriately. This completes the calibration form set up.

10.10 Acquire and Record Multipoint Analyzer Response Data (Y):
Remove the cells from the calibration bench and go to measurement mode and begin measurements.

Using the Multipoint Span/Offset Calibration Worksheet, record at least **three consecutive values** for the concentration and deviation reported by the analyzer with no cells in the bench.

Repeat this procedure for all other cell combinations indicated on the form and in section 10.9.5.

Since it takes some time to add or remove cells, at least one measurement cycle should be skipped to ensure that all cells are present for the full measurement cycle.

Average at least the three consecutive values for each cell combination and record the value in the space provided on the Multipoint Span/Offset Calibration Worksheet.

Transcribe the average concentration values and deviations from the Multipoint Span Calibration Worksheet to the Multipoint Span/Offset Calibration Form in columns (Y) Anal. Resp. (Column 5) and Stand. Dev. (Column 6).

10.11 Implement Nonlinear Correction:

Open the Non-Linear correction menu and activate benzene only. Enter the measured (Y) and expected (X) concentration values for 5 sets of data: The default selections are:

Point 1 (no cells)
Point 5 (cell L_{C3})
Point 6 (cells $L_{C1} + L_{C3}$)
Point 7 (cells $L_{C2} + L_{C3}$)
Point 8 (cells $L_{C1} + L_{C2} + L_{C3}$)

The nonlinear correction coefficients are calculated and applied as described in Section 5 (Pg 5-23) of the OPSIS Opto-Analyzer Hardware Operating Manual and Quality Assurance Manual. Press enter to accept the values. And exit the calibration submenu. Double check to make sure nonlinear correction is active. Note that implementation of a small offset (+/- 0.0 to 3 ppbV) in the "change span offset" can be used for optimization purposes. The "Span" setting with nonlinear correction active should always equal 1.0.

10.12 Verify Calibration:
Perform the 4-Point Calibration (Section 11) to verify the calibration of the system.

11.0 <u>4-point Calibration Check For Hg Measurements</u>:
The purpose of this procedure is to verify the calibration of the OPSIS system for Hg measurements. The operator must be familiar with procedures described in Section 10 prior to executing this procedure.

Good record keeping is essential and must be followed by the instrument operator. An analyzer-specific log book and hard copy calibration sheets must be maintained together with the instrument. For the procedure, the Span/Offset Calibration Check Form (Appendix C3) and the Multipoint Span/Offset Calibration Worksheet (Appendix C2) are used to record all calibration verification data.

11.1 Preparation for 4-point Calibration Check:
To prepare for the 4-point calibration check, execute all steps of Section 8, Section 9, and Section 10.0 through 10.7 with the following changes in preparation of the Span/Offset Calibration Check Form (Appendix C3):

<u>Span/Offset Calibration Check Form</u>:

· Fill-in the title section (top block) with analyzer and site information

· Fill-in the Calibration Setup Data
<u>Temperature</u>: This will be logged by the analyzer.
<u>Pressure:</u> This will be logged by the analyzer.
<u>Light Int.</u> (from analyzer in %): <u>Lux</u> 1,500 – 1,900 (See 10.5)
<u>GG 400?</u>: no,

LF215/220?: no

- Fill-in the Initial Analyzer Data Calibration Setup Data
  Monitoring Path Length, L: 250 m (see Table 1)
  Reference ID: Current Reference date (eg 051107)
  Emitter Lamp ID: B7
  Span Setting (existing): Record the current span setting (should be 1.0)
  Offset Setting (existing): Record the current offset setting

- Fill-in the Preparations block:
  Cal. Measurement time: 56 seconds
  Initial System Name: N/A
  System Name Changed: N/A
  Cal. System Name "SPAN.CAL": N/A

- Normal Path Measurement Time: 56 seconds

- Fill-in the System Check block: Use data from procedure 8.5

- Fill-in the Calibration Lamp CA 004 test: Use data from section 9.5

Multipoint Span/Offset Calibration Worksheet

- Fill-in the title section including the active reference file

- Fill-in the title Fiber and Lamp ID information

The Multipoint Span/Offset Calibration Worksheet will be used to record the raw data to be averaged for the final calibration record (Span/Offset Calibration Check form).

11.2 Prepare Instrument and Calculate Expected Response:

11.2.1 Measurement Parameter Setup Check:
The 4-point calibration check is made in measurement mode with no change in the operational parameters of the instrument. Go to the **Change span/offset** menu under **Calibration**, make a note of the existing span and offset values for path one and record these values. Ensure that nonlinear correction is active. The Span setting should be 1.0 in this case.

11.2.2 Temperature Correction Setup Check:
Go to the **Path specifications** menu under **Measurement setup**. Ensure that the NTP (temperature/pressure correction) value is 25C.

11.2.3 Note Calibration Start and Stop Time:
Since the analyzer will be operating in normal measurement mode during calibration, it is critical not to confuse normal measurement values with calibration values. Make sure to note the start and stop times for all calibration work in the analyzer log book so that calibration data can be properly segregated from actual measurement data during data validation and archiving.

11.2.4 Check and Record Lux Level:
Ensure that the reduced lux level of (default 1,500 – 1,900 lux) set in 10.4 is accurate and properly recorded in calibration setup data of the calibration form.

11.2.5 Insert Calibration Cells: Place the three cells in the bench and fill them with the reference gas standard.

11.2.6 Record the Expected Analyzer Response (X):
Calculate the expected response value of the instrument (X) for each of the cell combinations in 11.2.5 and record the value in column 4 of the Span/Offset Calibration Check Form adjacent to its respective total Cal. Cell Length (L_c). It is noted that (X) is based on (and equivalent to) a length corrected analyzer response (C_c') which is modified by the stored measurement path length (L). The measurement path length (L) is automatically present in all concentration calculation so must be decoupled appropriately. An example calculation follows:

$X = C_c' = C_c \times (L_c/L)$;

Where: C_c' = Length Corrected Analyzer Response (in ppbV)
C_c = Hg Cell Concentration (in ppbV)................................
L_c = Total Cal. Cell Length
L = Measurement path length

11.3 Acquire and Record Analyzer Response Data (Y):
Remove the cells from the calibration bench and go to measurement mode and begin measurements.

Using the Multipoint Span/Offset Calibration Worksheet, record at least **three consecutive values** for the concentration and deviation reported by the analyzer with no cells in the bench. This record should be entered in the bottom set of blocks on the worksheet.

Repeat this procedure for all other cell combinations indicated on the form and in section 11.2.5.

Since it takes some time to add or remove cells, at least one measurement cycle should be skipped to ensure that all cells are present for the full measurement cycle. The temperature of the cells should be periodically monitored and noted in the log book should not deviate from the original recorded temperature by more than +/- 0.1 degrees C. If the temperature varies more than this, then the temperature for each reading should be recorded and concentrations appropriately calculated and averaged.

Average at least the three consecutive values for each cell combination and record the value in the space provided on the Multipoint Span/Offset Calibration Worksheet.

Transcribe the average concentration values and deviations from the Multipoint Span/Offset Calibration Worksheet to the Span/Offset Calibration Check Form in columns (Y) Anal. Resp. (Column 5) and Stand. Dev. (Column 6).

11.4 Compare Results to Evaluate Data Quality Indicator Goals:

The expected and measured analyzer values (accuracy) should be within 15% for each of the three consecutive values determined from each of the three cell combinations. The accuracy of the instrument is calculated using the following formula:

$$\left[1-\left(\frac{\text{Measured Value}}{\text{Expected Value}}\right)\right]\times 100$$

The precision of the calibration values for each of the three cell combinations must be evaluated by assessing the variations (among the three values from each of the three cell combinations) of the results. Precision is determined by calculating the relative standard deviation (RSD) of each set of three measurements. The precision of each of the three sets of three values should fall within 15%.

If the calculated accuracy and precision of each measurement does not fall within 15%, repeat the Span/Offset Calibration Check to eliminate cell insertion error potential. If the calibration is still out of specification the multipoint span offset calibration and/or consult EPA OPSIS expert Cary Secrest for troubleshooting procedures.

* Note: The procedures used to calculate the minimum detection limit of the instrument can be found in Section 5.3 of Appendix E of this document.

12.0 Xenon Lamp Reference Check and New Lamp Reference Recording

Xenon lamp reference checks and new lamp reference spectrum recording are performed to measure and record changes in the (raw) source lamp spectrum. The lamp reference spectrum represents the background which the system subtracts from measurement spectra to minimize noise. This spectrum needs to be updated to compensate for changes in the lamp, analyzer spectrometer, and electronics.

Note: For this PSOP, a new lamp reference spectrum is recorded before initiation of the Multipoint Span/Offset Calibration to ensure that an optimized reference spectrum with the primary calibration lamp and instrument specific fibers is used. This reference spectrum must be taken a lux level of 3000 as measured after the 5 m fiber which connects to the analyzer. This reference spectrum should be used for the entire duration of the project. The recording of a new reference must be properly noted in the analyser notebook.

Good record keeping is essential and must be followed by the instrument operator. An analyzer-specific log book and hard copy calibration sheets must be maintained together with the instrument. For the procedure, the OPSIS Reference Check form (Appendix C4) is used to record all calibration data.

12.1 Preparation for Reference Check:
The standard setup is shown schematically in Figure 5. Procedures of Sections 8, 9 and 10.0 through 10.7 must proceed this operation. Ensure that the lamp has at least 30 minutes warm-up time for it to stabilize.

Prepare the Reference Check form in a similar manner to that described in previous sections.

12.2 Software Setup:
Using the installation menu, select **Calibration setup**. Go to **Calibration** path setup, and set the parameters as follows:

- Physical path 1
- Path control Manual

The other parameters are not used in manual calibration.

Reference calibration setup parameters can be checked by going to the **Reference calibration setup** menu. Set the key parameters as follows:

- Active Yes
- Calibration path 1
- Measurement time 300 seconds

The other parameters are not used in manual calibration.

12.3 Reference Check Performance Procedure

The following procedure establishes the viability of the currently stored reference spectrum.

12.3.1 Fiber and Lamp ID Check:

Check the current field log to ensure the correct lamp, and fibre optic cable has been installed in the optical calibration bench.

12.3.2 Note Calibration Start and Stop Time:

Since the analyzer will be operating in normal measurement mode during calibration, it is critical not to confuse normal measurement values with calibration values. Make sure to note the start and stop times for all calibration work in the analyzer log book so that calibration data can be properly segregated from actual measurement data during data validation and archiving.

12.3.3 Reference Check:

A number of test measurements will be made using the first measurement path. Go to the **Change span/offset** menu. Under **Calibrations**, make a note of the initial offset value for path one for the gases to be checked, and set these offsets to a zero.

Go to **Measurement time**, and set the time according for benzene to 56 seconds (Table 1). Set the measurement time for all other gases to zero.

Set the analyzer to measurement mode. Let the analyzer measure for four complete cycles, and note the concentrations, deviations, and light levels being reported.

Go to **Calibration** and select **Reference calibration**. Set **Skip reference check** to **Yes**, and mark all gases currently under investigation. Then select **Start calibration**. Proceed through the reference recording for all gases (Hg only). There is now a new reference spectrum for benzene.

Go back to measurement mode and complete another four cycles, making note of the results. These are measurements using the new reference spectra.

Study the deviations for benzene obtained with the old and the new reference spectra. If the new deviations are of equal or smaller range, then the new reference should be used. The old reference should therefore NOT be restored. However, if the deviations have increased, or the concentrations changed drastically, the new reference is actually worse than the old reference spectra. This should normally not occur, but might happen if something is wrong with the reference check setup. The new reference should then not be used. Instead, restore the old one by going to the **Restore reference** function in the **Calibration** menu. Select the gas, and press **[P]** to restore the previous (i.e., old) reference spectrum.

13.0 Disk Space and Analyzer Clock Check

13.1 The disk space will remain acceptable over the duration of the project, as data files will be downloaded on a daily basis, and the analyzer hard drive contains sufficient disk space for many months of data acquisition.

13.2 The analyzer clock shall be reset using the analyzer on-screen directions if necessary to synchronize with the reference time used by the DIAL. The time offset shall be less than 60 seconds.

14.0 References

Opto-Anlayser AR500 for Air Quality Monitoring, Hardware Operating Manual. OPSIS. March 1995.

Standard Operating Procedure for Analyzer Field Operation. Revision No. 0. December 2000.

OPSIS Analyzer Software User's Guide. Version 7.2, May 1999.

OPSIS Qaulity Assurance and Quality Control using Opsis Analyser for Air Quality Monitoring. Version 1.4, February 2003.

APPCD's OPSIS Safety Protocol. November 2005.

Table 1. Standard Calibration Set-up Parameters

Calibration Gas	Path Length (m)	Measurement Time (seconds)
benzene	250 m	56

Table 2. Calibration and Operational Check Schedule for Benzene Opsis PSOP

Group	Check/Test	Frequency
Calibration	Eight-Point Span/Offset Calibration	Pre-deployment, or when indicated necessary by the four-point calibration check
QC Check	Four-Point Calibration Check	Pre-deployment, and at three-week intervals
QC Check	Wavelength Precision Check	Pre-deployment, and at three-week intervals
QC Check	System Check	Pre-deployment, at time of initial deployment in the field, and daily during telemetry download
QC Check	Disk Space and Analyzer Check	Initial deployment in the field, and daily during measurement.
QC Check	Reference Check	Pre-deployment

Figure 1. Calibration Bench Connection to Lux Meter

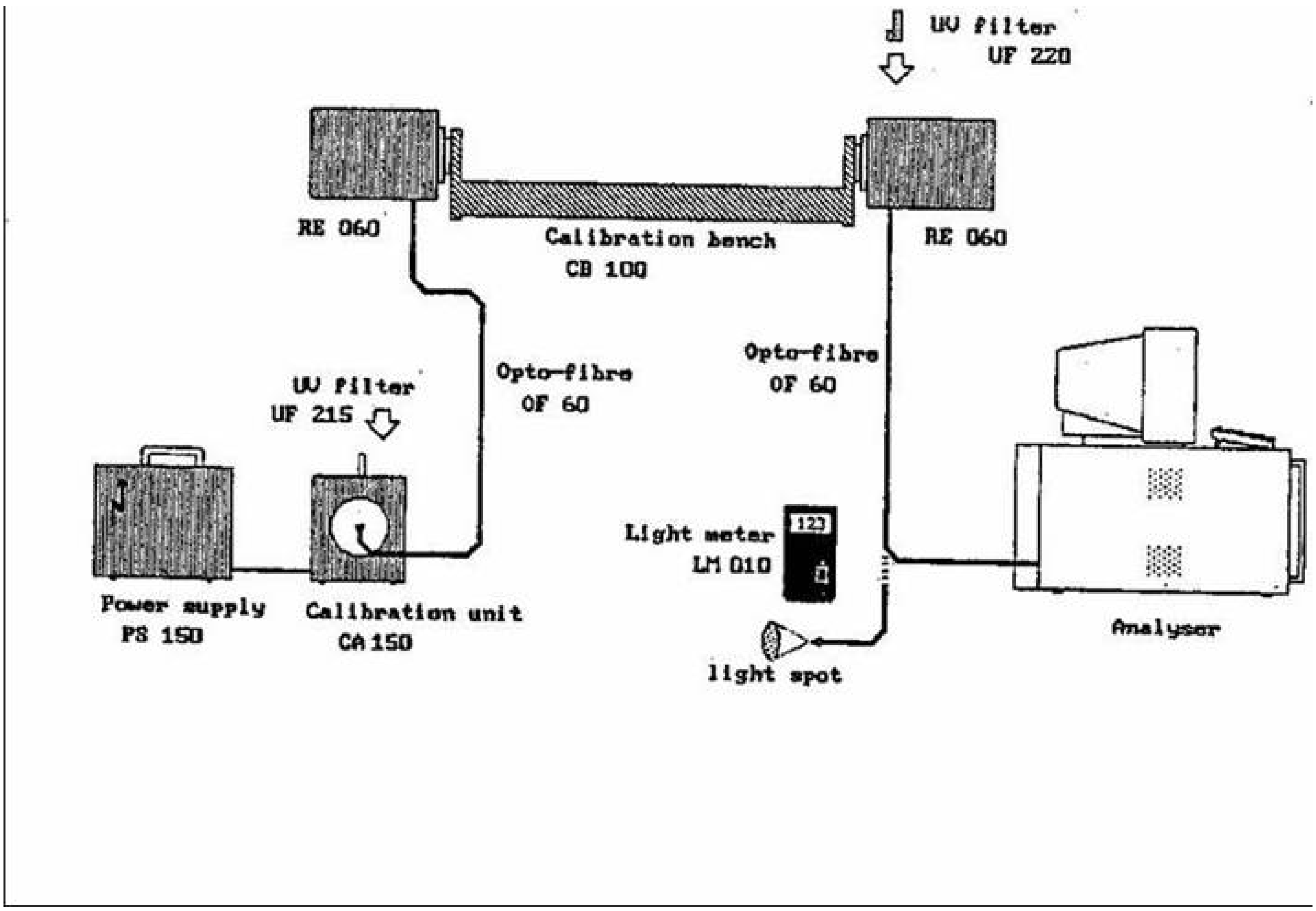

Figure 2. Standard Calibration Bench Setup

Appendix C1. Multipoint Span/Offset Calibration Form

Opsis Analyzer for Air Quality Monitoring

MULTIPOINT SPAN/OFFSET CALIBRATION

Analyzer S/N E488 Site Location ______________ Date ______________

Gas Cylinder ID ______________ Test Gas ______________ Last Calibration ______________

2

Calibration Setup Data

Cell length(s) L_c........ L_{c1} = 0.1006 m
L_{c2} = 0.2488 m
L_{c3} = 0.5015 m

Temperature........ ______________
Pressure........ ______________
Light Int..... ______________ Lux.. ______________
GG 400?........ Yes ☐ No ☐
LF 215/220?........ Yes ☐ No ☐

3

Initial Analyzer Data

Reference path length L........ ______________
Reference ID........ ______________
Emitter lamp ID........ ______________
Span setting (existing)........ ______________
Offset setting (existing)........ ______________

4

Normal Path Measurement Time ______________

5

Preparations

Cal. Measurement Time........ ______________
Initial System Name........ ______________
System Name Changed........ ok ☐
Cal. System Name "SPANCAL"..... ok ☐

6

System check
P1 ______________
P2 ______________
P3 ______________
P4 ______________
P5 ______________

All ok?
Yes ☐ No ☐
Repeated?
Yes ☐ No ☐
Correction P4?
Yes ☐ No ☐

Final setting
P1 ______________
P2 ______________
P3 ______________
P4 ______________
P5 ______________

7

Calibration lamp CA 004 test

Shift........ ______________ ch.
Adjusted..... Yes ☐ No ☐
Final shift... ______________ ch.

8

	Cal. Gas conc. C/ppm	Cell conc. C_c/ppm	Cal cell length L_{cal}	(X) Length Correction $C_c' = (C_c) \times (L_c/L) $ / ppb	(Y) Analyzer resp. / ppb	Standard dev. / ppb
			0.0000			
L_{c1}			0.1006			
L_{c2}			0.2488			
$L_{c1} + L_{c2}$			0.3494			
L_{c3}			0.5015			
$L_{c1} + L_{c3}$			0.6021			
$L_{c2} + L_{c3}$			0.7503			
$L_{c1} + L_{c2} + L_{c3}$			0.8509			

Linear regression
Y = k(X) + b

k = ________
b = ________
r = ________

Final settings: Span ______________
Offset ______________

9

Operator comments

10

____
Operator Signature

Appendix C2. Multipoint Span/Offset Calibration Worksheet

Multipoint Span Calibration

Date:__________________ Operator:________________________

Component Reference No.:__________________

Bench Configuration		1	2	3	4	5	6	7	8	Average
0	Val									
	Std									
1	Val									
	Std									
2	Val									
	Std									
1+2	Val									
	Std									
3	Val									
	Std									
1+3	Val									
	Std									
2+3	Val									
	Std									
1+2+3	Val									
	Std									
Check										
1	Val									
	Std									
1+2	Val									
	Std									
1+2+3	Val									
	Std									

Fiber Record

Cal Lamp ____________________ Hg Lamp ____________________

Cal Bench ____________________ Outside Rec.____________________

Appendix C3. Span/Offset Calibration Check Form

Opsis Analyzer for Air Quality Monitoring

SPAN/OFFSET CALIBRATION CHECK

Analyzer S/N E466 ______ Site Location ______ Date ______

Gas Cylinder ID ______ Test Gas ______ Last Calibration ______

2

Calibration Setup Data

- Cell length(s) L_c........ L_{c1} = 0.1006 m
- L_{c2} = 0.2488 m
- L_{c3} = 0.5015 m
- Temperature........ ______
- Pressure........ ______
- Light Int...... ______ Lux.. ______
- GG 400?........ Yes ☐ No ☐
- LF 215/220?........ Yes ☐ No ☐

3

Initial Analyzer Data

- Reference path length L........ ______
- Reference ID........ ______
- Emitter lamp ID........ ______
- Span setting (existing)........ ______
- Offset setting (existing)........ ______

4

Normal Path Measurement Time ______

5

Preparations

- Cal. Measurement Time........ ______
- Initial System Name........ ______
- System Name Changed........ ok ☐
- Cal. System Name "SPAN.CAL"........ ok ☐

6

System check

- P1 ______
- P2 ______
- P3 ______
- P4 ______
- P5 ______

All ok?

- Yes ☐ No ☐

Repeated?

- Yes ☐ No ☐

Correction P4?

- Yes ☐ No ☐

Final setting

- P1 ______
- P2 ______
- P3 ______
- P4 ______
- P5 ______

7

Calibration lamp CA 004 test

- Shift........ ______ ch.
- Adjusted.... Yes ☐ No ☐
- Final shift.... ______ ch.

8

Cal. Gas conc. C/ppm	Cell conc. C_c/ppm	Cal cell length L_{cal}	(X) Length Correction $C_c' = (C_c) \times (L_c/L)$ / ppb	(Y) Analyzer resp. / ppb	Standard dev. / ppb
		0.0000			
L_{c1}		0.1006			
$L_{c1} + L_{c2}$		0.3494			
$L_{c1} + L_{c2} + L_{c3}$		0.8509			

r =

Final settings: Span ______ Offset ______

9

Operator comments

10

______

Operator Signature

Appendix C4. Reference Check Form

Opsis Analyser for Air Quality Monitoring

REFERENCE CHECK

Analyser S/N E466 Site Location ______ Last Calibration ______ Date ______

2

System check | All ok? | Final setting
P1 ______ | Yes ☐ No ☐ | P1 ______
P2 ______ | Repeated? | P2 ______
P3 ______ | Yes ☐ No ☐ | P3 ______
P4 ______ | Correction P4? | P4 ______
P5 ______ | Yes ☐ No ☐ | P5 ______

3

Wavelength Precision	Shift	Adjust y	Adjust n	Final Shift
gas ______	______	☐	☐	______
gas ______	______	☐	☐	______
gas ______	______	☐	☐	______
gas ______	______	☐	☐	______
gas ______	______	☐	☐	______

4

Reference light spot.................. ok ☐
Reference light intensity.......... ______
Cal. Lamp ID.......................... ______ Lux ______

5

Initial System Name.................. ______
System Name Changed?............. ok ☐
Ref. System Name "SPAN.CAL"...... yes ☐

6

Reference check and recording

Gas name..........	______	______	______	______	______
Unit................	______	______	______	______	______
Lamp type..........	A ☐ B ☐	A ☐ B ☐	A ☐ B ☐	A ☐ B ☐	A ☐ B ☐
UF in use?..........	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐
Original offset......	______	______	______	______	______
Offset zero?.........	ok ☐	ok ☐	ok ☐	ok ☐	ok ☐
Std. meas. time.....	___:___	___:___	___:___	___:___	___:___
Measurement time	___:___	___:___	___:___	___:___	___:___
Light level..........	______ %	______ %	______ %	______ %	______ %
Reference ID........	______	______	______	______	______
Pre conc, dev1......	___.___	___.___	___.___	___.___	___.___
Pre conc, dev2......	___.___	___.___	___.___	___.___	___.___
Pre conc, dev3......	___.___	___.___	___.___	___.___	___.___
Pre conc, dev4......	___.___	___.___	___.___	___.___	___.___
Post conc, dev1.....	___.___	___.___	___.___	___.___	___.___
Post conc, dev2.....	___.___	___.___	___.___	___.___	___.___
Post conc, dev3.....	___.___	___.___	___.___	___.___	___.___
Post conc, dev4.....	___.___	___.___	___.___	___.___	___.___
New reference req.?	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐
Old reference rstrd?	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐	yes ☐ no ☐

7

Offsets restored?........................ ok ☐
Reasonable measurement results?... ok ☐

8

Operator Comments

9

____
Operator Signature